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IS 10132 (1982): Method of Test for Materials for Use in the Preparation of Magnesium Oxychloride Flooring Compositions [CED 5: Flooring, Wall Finishing and Roofing]



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IS : 10132 - 1982

Indian Standard

METHOD OF TEST FOR MATERIALS FOR USE IN THE PREPARATION OF MAGNESIUM OXYCHLORIDE FLOORING COMPOSITIONS

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Indian Standard

METHOD OF TEST FOR MATERIALS FOR USE IN THE PREPARATION OF MAGNESIUM OXYCHLORIDE FLOORING COMPOSITIONS

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Indian Standard

METHOD OF TEST FOR MATERIALS FOR USE IN THE PREPARATION OF MAGNESIUM OXYCHLORIDE FLOORING COMPOSITIONS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 February 1982, after the draft finalized by the Flooring and Plastering Sectional Committee, had been approved by the Civil Engineering Division Council.

0.2 Hitherto, methods of tests to assess the qualitative requirements of materials for use in the manufacture of magnesium oxychloride flooring compositions were included in IS : 657-1960*. For facilitating the use, these methods of tests have been separated and covered in this standard.

0.2.1 While formulating this Indian Standard, the actual practice being followed by different testing authorities in the country has been kept in view. The procedure to determine the moisture content of fillers has been added and method of calculating the percentage of resin content in fillers has been modified. A method to analyze the fineness of calcined magnesite has also been included and preparation of standard test mixes as well as determination of standard consistency of test mixes for use in the tests of setting time, modulus of rupture and linear change of calcined magnesite has been standardized.

0.3 In reporting the results of a test or analysis in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard covers the methods of tests for determination of the following properties of calcined magnesite and fillers used in the preparation of magnesium oxychloride flooring compositions.

*Specification for materials for use in the manufacture of magnesium oxychloride flooring composition (*first revision*).

†Rules for rounding off numerical values (*revised*).

<i>Tests</i>	<i>Clause Ref</i>
a) Carbon dioxide	2
b) Fineness	3
c) Setting time	4
d) Modulus of rupture	5
e) Linear change	6
f) Bulk density	7
g) Resin content	8
h) Moisture content	9
j) Hardness	10
k) Compressive strength	11

2. CARBON DIOXIDE

2.1 Object — To determine the percentage of carbon dioxide present in calcined magnesite.

2.2 Apparatus — The arrangement, as shown in Fig. 1, shall be assembled with the following apparatus.

2.2.1 U-tube *A* with anhydrous granulated calcium chloride.

2.2.2 U-tube *B* with soda asbestos.

2.2.3 Funnel *C* with a glass stopcock *S* and a bent inlet glass tube *D* (projecting below the acid level) to introduce hydrochloric acid into the generating flask.

2.2.4 Condenser *E* for condensing most of the water and hydrochloric acid.

2.2.5 U-tube *F* containing zinc pellets to react with residual acid, if any.

2.2.6 Scrubber *G* containing concentrated sulphuric acid to absorb moisture.

2.2.7 U-tube *H* containing pumice stone, impregnated with anhydrous copper sulphate to remove traces of hydrogen sulphide. This is prepared by crushing pumice stone to approximately 5 mm size, sifting it free from dust and then transferring 60 g of it to a cassorole. It is then covered with a concentrated solution of 30 to 35 g of copper sulphate and then evaporated to dryness while stirring constantly. It is then heated for 3 to 4 hours at 150 to 160°C corked in a desiccator and preserved in a glass-stoppered bottle.

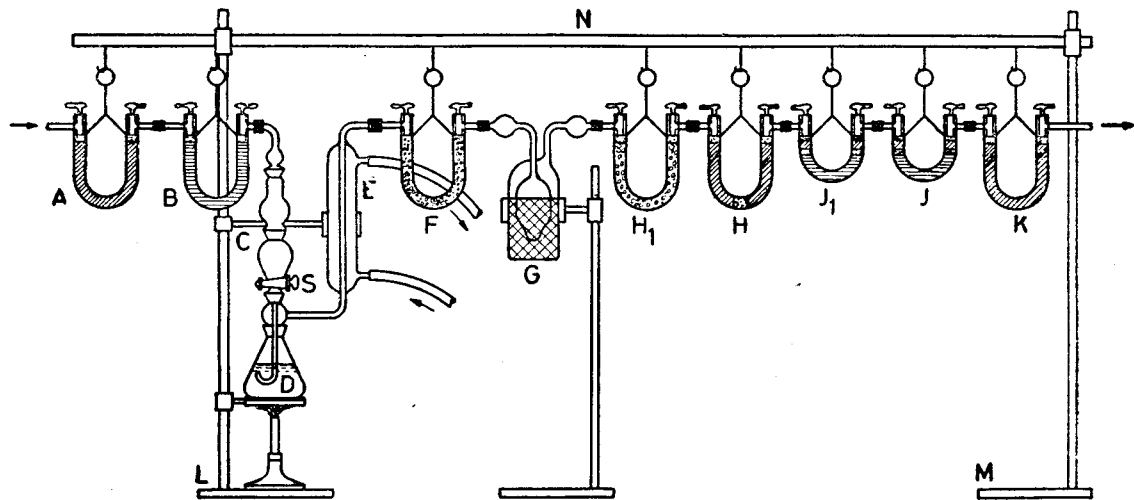


FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF CARBON DIOXIDE IN MAGNESITE

2.2.8 U-tube *H* containing anhydrous granulated calcium chloride in one limb and anhydrous magnesium perchlorate in the other to remove the last traces of moisture.

2.2.9 U-tube \bar{J}_1 and \bar{J} containing both soda asbestos and anhydrous magnesium perchlorate in each limb to absorb carbon dioxide.

2.2.10 U-tube *K* containing anhydrous granulated calcium chloride and anhydrous magnesium perchlorate in each limb to protect the end of the train against moisture.

2.2.11 A cross-bar *N* for suspending the U-tubes.

2.2.12 Retort stands *L* and *M* for supporting the cross-bar *N*.

2.2.13 All joints are made of stout-walled rubber tubing with the ends of the glass tubes touching each other. Means are provided to pass air through the system either under pressure or by suction.

2.3 Reagent

2.3.1 *Dilute Hydrochloric Acid (AR)* — It is prepared by diluting hydrochloric acid (sp gr 1.16) with distilled water in the ratio of 1 : 4 by volume.

2.4 Samples — Suitable quantity of fresh sample shall be taken.

2.5 Procedure

2.5.1 Weigh accurately about 2.5 g of the sample, transfer it into the flask *D* and add 20 to 40 ml of distilled water to it. Insert the glass stopper carrying the funnel arrangement and the condenser to make the apparatus airtight. Pass air through the system until the carbon dioxide absorption tubes attain constant weight. Close the stopcock *S* and replace the weighed carbon dioxide absorption tube \bar{J}_1 and \bar{J} in the system. Introduce 40 ml of hydrochloric acid (1 : 4) into the flask *D* through the funnel *C* and heat the flask *D* carefully so that the gases may pass through the sulphuric acid bubbler at moderate rate. After about 30 to 40 minutes, boil the contents of the flask. After two or three minutes of boiling, remove the flame and connect the flask to scrubber *A* and *B* by means of stopcock *S*, and pass air through the system for 20 minutes at a moderate rate. Keep the absorption tubes \bar{J}_1 and \bar{J} at room temperature for 30 minutes and then weigh. The increase in weight denotes the carbon dioxide content in the sample.

2.6 Report — Report the content of carbon dioxide as a percentage of the sample.

3. FINENESS

3.1 Object — To determine the particle size of calcined magnesite.

3.2 Apparatus and Reagent

3.2.1 IS sieves of nominal aperture size 150 and 75 μm [see IS : 460 (Part I)-1978]*.

3.2.2 An oven capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$.

3.2.3 *Methylated Spirit (AR)*

3.3 Sample — A fresh sample of calcined magnesite weighing 10 to 12 g.

3.4 Procedure — Weigh and place a 150 μm IS sieve, 75 mm in effective diameter of sieving surface in a dish. Place the weighed sample in the sieve and gently pour methylated spirit (recently dried over fresh plaster of paris) over the sample until the gauze is covered. Perform the sieving by moving the sieve up and down in the liquid for 5 minutes gently tapping the side of the sieve, stirring the sample and the liquid from time to time with a soft camel hair pencil brush. Add more dried methylated spirit, if necessary. Raise the sieve from the liquid and wash the sample with dried methylated spirit until the washings are clear. Dry the sieve and its contents in an oven at $105 \pm 2^\circ\text{C}$ for one hour and weigh. Transfer the liquid containing in suspension the portion of the sample which has passed the 150 μm IS sieve to a weighed 75 μm IS sieve, 75 mm in effective diameter standing in another dish. Repeat the sieving operation as above for 15 minutes and finally wash the sieve and its contents, dry and weigh as before.

NOTE — Mechanical sieving can be done provided care is taken to ensure that breaking up of particles does not occur and that sieving is complete.

3.5 Report — The weights of the two residues are determined and percentage passing through 150 μm IS sieve and 75 μm IS sieve shall be reported.

4. SETTING TIME

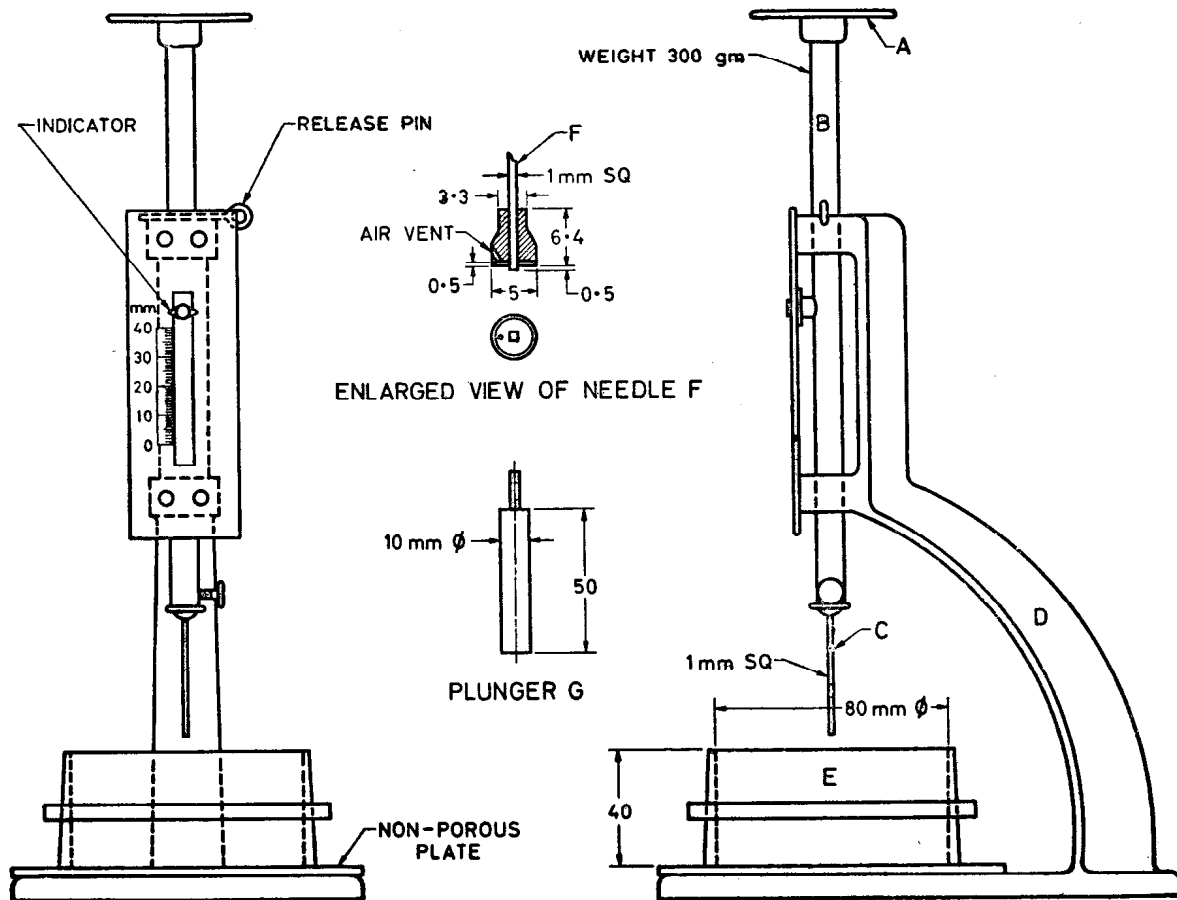
4.1 Object — To determine the initial and final setting time of the standard consistency paste of calcined magnesite.

4.2 Apparatus — A Vicat apparatus as illustrated in Fig. 2.

4.2.1 The apparatus consists of a frame *D* bearing a movable rod *B* with the cap *A* at one end and at the other, one of the following which are removable:

- a) The needle *C* for determining the initial setting time,

*Specification for test sieves: Part I Wire cloth test sieves (*second revision*).



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All dimensions in millimetres.

FIG. 2 ENLARGED VIEW OF NEEDLE F

- b) The needle *F* for determining the final setting time, and
- c) The plunger *G* for determining the normal consistency.

4.2.1.1 The needle *C* shall be 1 mm² or 1.13 mm diameter in section and have a flat end. The needle *F* shall be of the same shape and section as needle *C* but shall be fitted with a metal attachment hollowed out so as to leave a circular cutting edge 5 mm in diameter, the end of the needle projecting 0.5 mm beyond this edge. The plunger *G* shall be of polished brass 10 mm in diameter, 50 mm long and with a projection at the upper end for insertion into the movable rod *B*. Its lower edge shall be flat. The movable rod *B* carries an indicator which moves over a graduated scale attached to the frame *D*.

4.2.1.2 With all attachments, the cap and the rod with needle *C* (or needle *F*, or plunger *G*) shall together weigh 300 g.

4.2.1.3 The mould consists of a split ring 80 mm in diameter and 40 mm high which rests on a non-absorbent plate.

4.3 Procedure

4.3.1 Prepare a test block by mixing 3 parts by weight of calcined magnesite with 1 part by weight of wood flour to the standard consistency described in Appendix A.

4.3.1.1 During the test, keep the test block at a temperature of $27 \pm 2^\circ\text{C}$ and in an atmosphere of at least 90 percent relative humidity and away from draughts.

NOTE — An atmosphere of over 90 percent relative humidity can be attained by covering the specimens with a damp cloth which, however, should not be allowed to come into contact with the specimens themselves.

4.4 Determination of Initial Setting Time

4.4.1 Place the test block confined in the mould *E* and resting on the plate under the rod bearing the needle *C*; lower the needle gently into contact with the surface of the test block and quickly release allowing it to sink into the test block. Repeat this process until the needle, when brought into contact with test block and released as described above, does not penetrate to the base. The material shall then be regarded as having attained its initial set, and the time which has elapsed since the magnesium chloride solution was added to the dry ingredients shall be taken as the initial setting time.

4.5 Determination of Final Setting Time

4.5.1 Replace the needle *C* of the Vicat apparatus by the needle *F*. The paste shall be considered as finally set when, upon and applying the

needle gently to the surface of the test block, the needle makes an impression thereon, while the attachment *F* fails to do so, and the time which has elapsed since the magnesium chloride was added, shall be taken as the final setting time.

5. MODULUS OF RUPTURE

5.1 Object — To assess the modulus of rupture of a standard consistency paste of calcined magnesite.

5.2 Samples

5.2.1 The transverse strength of calcined magnesite shall be ascertained from tests on beams measuring $100 \times 25 \times 25$ mm prepared and cured in the manner as given under 5.2.2 to 5.2.4.

5.2.2 Preparation of Beams — Prepare the beams by mixing 3 parts by weight of calcined magnesite and one part by weight of wood flour to standard consistency described in Appendix A. The size of moulds shall be the size of beams.

5.2.3 Place each metallic mould of internal dimensions $100 \times 25 \times 25$ mm horizontally on a non-absorbent plate and fill them with the mixture as gauged above and consolidate the same with finger pressure only. Finish the surface of the test pieces smooth with the blade of a trowel or plate knife. The mixture is filled into the metal moulds after lightly greasing them with petroleum jelly.

5.2.4 Prepare 12 beams as above, keep at $27 \pm 2^\circ\text{C}$ in an atmosphere of at least 90 percent relative humidity for 24 hours. After gauging, remove them from the moulds and immediately place them in a tray covered with not less than 25 mm depth of dry sand. Store the trays containing the test pieces away from draughts in a room maintained at a temperature of $27 \pm 2^\circ\text{C}$ and relative humidity 65 to 70 percent.

NOTE — An atmosphere of 65 to 70 percent relative humidity can be attained by placing a tray containing solid ammonium nitrate in the vessel containing the specimens, the vessel being kept closed and the air within it circulated by means of a fan. If the solid ammonium nitrate gets covered with liquid, the tray must be emptied and replenished.

5.3 Procedure

5.3.1 Rest the beam to be tested with one side on two parallel metal rollers 12.5 mm in diameter and placed apart at 75 mm centres. Apply the load to the opposite side at a uniform rate of 25 kg per minute through a third similar roller placed midway between the other two. Do not use any packing between the rollers and the test piece.

5.3.2 For determining the breaking load at 168 ± 2 h (7 days) and 672 ± 4 h (28 days) test 6 beams as specified under 5.3.1 after the specific periods after gauging the mix with magnesium chloride solution.

5.4 Calculation

5.4.1 The modulus of rupture shall be computed from the following formula:

$$R = 0.72 W$$

where

R = modulus of rupture in kg/cm², and

W = breaking load in kg.

5.5 Report

5.5.1 The average of six determinations at 7 days and 28 days shall be reported.

6. LINEAR CHANGE

6.1 **Object** — To calculate the change in length of a standard consistency paste of calcined magnesite, the linear change shall be determined on beams measuring 200 × 25 × 25 mm or of any other size to suit the apparatus available, cast in metal moulds, each mould or set of moulds resting on a non-absorbent plate. Both the mould and the plate shall be lightly greased with petroleum jelly.

6.2 Number of Test Pieces

6.2.1 Four test pieces shall be prepared from the standard consistency paste.

6.3 Preparation of Test Pieces

6.3.1 The test pieces shall be prepared from standard-consistency paste described in Appendix A. The size of moulds shall be as described in 6.1.

6.3.2 The moulds shall be filled with paste and the surface shall be struck off so as to be in level with the top of the mould.

6.3.3 In filling the moulds use only hands and the blade of ordinary gauging trowel.

6.3.4 Cover the moulds with non-absorbent plate and transfer them without delay to the place of storage.

6.4 Storage

6.4.1 Allow the test pieces to remain in their moulds on non-absorbent plates and cover them with a wet cloth for a period of 24 hours.

6.4.2 Remove the test pieces from their moulds and measure their lengths accurately (see 6.5.3). Place the test pieces then on racks in a cupboard or container, each beam being supported on two glass rods 6 mm in diameter and placed at a distance of not less than 25 mm from the neighbouring beams. Maintain the air in the cupboard or container at $27 \pm 2^\circ\text{C}$ and at a relative humidity of 65 to 70 percent.

NOTE — Certain bottom-coat mixes may be too friable to be handled with safety after 24 hours. These may be left in their moulds for 48 hours and the fact recorded. If after 48 hours the specimen still cannot be removed from the moulds intact, the mix shall be deemed to have failed.

6.5 Measurement

6.5.1 Measure the length of the specimens at 672 ± 4 hours (28 days) to the nearest millimetre.

6.5.2 Express the change in length (contraction or expansion) as a percentage of length at 24 hours age.

6.5.3 Adopt any convenient method of measurement which enables changes in length of the order of $1/40$ mm to be observed with accuracy. Two such methods, recommended for this purpose, are given below:

- a) Measure the distance between two reference points, for example, gramophone needles placed 6.5 to 9.5 mm from each end of the specimen, by means of a measuring microscope.
- b) Partially embed two steel balls 6.5 mm in diameter, one at each end of the specimens by placing them in a countersunk hole in a brass plate 2.5 cm square and placed upright at each end of the mould. The balls may be lightly held by a spot of petroleum jelly. When the mix has set and the beams are removed from the moulds, the brass plates can be separated from the moulds, from the ends of the beam, leaving the balls half-embedded in the mix. The distance between the balls is measured by means of a micrometer gauge.

NOTE — The method (b) is suitable only when the mix is of a type which sets hard in 24 hours, and is not generally suitable for bottom-coat mixes.

7. BULK DENSITY

7.1 Object — To calculate the bulk density of calcined magnesite expressed in terms of kg/litre.

7.2 Apparatus

7.2.1 The apparatus assembled as shown in Fig. 3 shall consist of a funnel *F*, containing a screen *S* (IS Sieve 3.35 mm) suspended above a cylindrical measure *M* of one-litre capacity of 100 mm internal diameter and 127 mm internal height, and a wooden spatula.

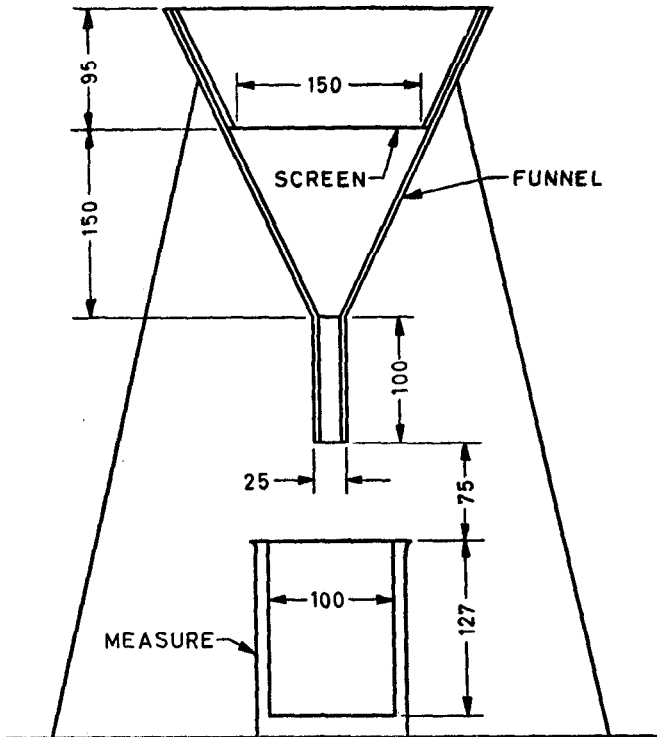
7.3 Procedure

7.3.1 Weigh the measure *M* and place it in position below the funnel *F*. Then place a sample of the calcined magnesite weighing about 1 kg on the screen and stir with the spatula until the measure *M* is filled and an excess of magnesite lies on top. Allow the measure to stand untouched for one minute. Without knocking the measure, strike off the surface of the magnesite making it level with the top of the measure.

Weigh the measure and its contents together and deduct the weight of the measure.

7.4 Report

7.4.1 The difference in weight shall be reported as the bulk density.



All dimensions in millimetres.

FIG. 3 ASSEMBLY OF APPARATUS FOR DETERMINING DENSITY OF CALCINED MAGNESITE

8. RESIN CONTENT OF FILLERS

8.1 Object — To calculate the percentage of resin content present in the fillers.

8.2 Procedure

8.2.1 Place 2 g of a dry wood flour or saw dust in a fused alumina filter crucible of 42 mm top external diameter, 45 mm in height and of porosity equivalent to Grade 4 and then weigh in a glass weighing bottle with ground in lid. Transfer the crucible with contents to a Soxhlet apparatus where extraction with 2 parts by volume of toluene and 1 part of ethyl alcohol, 95 percent by weight is carried on for 6 to 8 hours. Remove excess solvent by suction using either a glass funnel with straight sides and a rubber gasket or a 60° glass funnel with a vulcanite filtering crucible holder provided with a rubber flange, and rinse the contents of the crucible thoroughly with diethyl ether to remove all traces of alcohol and toluene. Remove excess ether by suction and dry the crucible and the contents to a constant weight in the original weighing bottle at a temperature of $105 \pm 2^\circ\text{C}$. The difference between the original and the final weights represents the combined resin and moisture content.

8.2.1.1 The moisture content is determined separately on a 2 g sample by drying in an oven at a temperature of $105 \pm 2^\circ\text{C}$ until no further loss occurs.

8.2.1.2 The resin content is found by difference as a percentage of the oven-dried material.

NOTE — If the weight of residue left after extracting 2 g of the sample is X g and if the weight of the material remaining after oven drying 2 g of the sample is D g, then the percentage of resin in the oven-dried material will be equal to

$$100 \times \frac{D - X}{100}$$

9. MOISTURE CONTENT OF FILLERS

9.1 Object — To calculate the percentage of moisture content present in the fillers.

9.2 Procedure — Dry 2 g of the filler to constant weight by heating in an oven at a temperature of $105 \pm 2^\circ\text{C}$.

9.2.1 Express the loss in weight as a percentage of the original weight of the sample.

10. HARDNESS

10.1 Object — To determine the hardness of the magnesium oxychloride floor finish.

10.2 Procedure — The hardness of the floor finish shall be compared with that of the pattern sample or other agreed surface in the manner given under **10.2.1** to **10.2.3**.

10.2.1 A sheet of handwriting carbon paper (typewriting carbon paper is not suitable) should be laid on the floor finish with the carbon face uppermost and covered with a thin sheet of white paper.

10.2.1.1 A steel ball, 25 mm diameter and weighing approximately 65 g, should then be dropped on to the paper from a height of 2 m.

10.2.1.2 The diameter of the impression (approx. to a circle) so produced on the underside of the white paper should be measured by means of a micrometer microscope or other convenient instrument reading to 0.1 mm. Readings should be taken in two directions at right angles and the mean value determined. Any impression which is obviously irregular in shape should be neglected.

10.2.1.3 The test should be made at six agreed positions on the floor surface and the mean diameter obtained.

10.2.2 The pattern sample or other agreed surface should be similarly tested.

10.2.3 The hardness of the floor finish should be deemed satisfactory if the mean diameter in test **10.2.1** does not differ from the mean diameter in test **10.2.2** by more than 0.5 mm.

11. COMPRESSIVE STRENGTH

11.1 Object — To determine the compressive strength of standard consistency paste of calcined magnesite.

11.2 Apparatus

11.2.1 Metal cubes of 70.6 mm sides.

11.2.2 Compression testing machine.

11.3 Samples

11.3.1 The compressive strength of calcined magnesite shall be ascertained from tests on cubes of size 70.6 mm prepared and cured in the manner given under **11.3.2** to **11.3.3**.

11.3.2 Prepare the cubes by mixing 3 parts by weight of calcined magnesite to 1 part by weight of wood flour to standard consistency described in Appendix A. The size of mould shall be as given in **11.3.1**.

11.3.2.1 Place each cubes on a nonabsorbent plate and fill them with the mixture as gauged above and consolidate the same with light

pressure only. Finish the surface smooth and place with the blade of a trowel or plate knife.

11.3.3 Prepare 6 cubes as above, and keep them at $27 \pm 2^\circ\text{C}$ in an atmosphere of at least 90 percent relative humidity for 24 hours. After gauging remove them from the moulds and place them in a tray. Store the trays away from draughts in a room maintained at a temperature of $27 \pm 2^\circ\text{C}$ and relative humidity of 65 to 70 percent.

11.4 Procedure — Test six cubes for compressive strength at the end of storing period in the compression testing machine.

11.5 Report — The compressive strength shall be calculated from the breaking load and the average area over which the load is applied. The average of all the determinations shall be reported in kg/cm^2 .

APPENDIX A

(*Clauses 4.3.1, 5.2.2, 6.3.1 and 11.3.2*)

PREPARATION OF STANDARD CONSISTENCY PASTE OF CALCINED MAGNESITE

A-1. Calcined magnesite and wood flour mixes of standard consistence are used in the tests for setting time, transverse strength and change of length.

A-2. Apparatus — For the determination of standard consistence, the Vicat apparatus illustrated and described in Fig. 2 is used, the plunger (G) 10 mm in diameter being substituted for the needle there shown in position.

A-3. Method

A-3.1 Mixing, Gauging and Filling Mould — A test mix shall be made by mixing 3 parts by weight of the calcined magnesite to 1 part by weight of wood flour (*see IS : 657-1982**). The dry materials are first thoroughly mixed together and then gauged with magnesium chloride of $22^\circ \text{Be}'$ [sp gr (d) = 1.179], on a non-absorbent slab. The mix is gauged with magnesium chloride solution of $22^\circ \text{Be}'$ [sp gr (d) = 1.179] to a consistency such that, when tested in the manner described below the Vicat plunger comes to rest at a distance not exceeding 10 mm from the bottom of the mould.

*Specification for materials for use in the manufacture of magnesium oxychloride flooring composition (*second revision*).

The time of gauging, that is the time elapsing from the moment of adding the solution to the dry calcined magnesite until commencing to fill the mould is not less than 4 minutes nor more than 6 minutes, during which time the mix is continuously worked. The mix is filled into the Vicat mould (*E*) (see Fig. 2), the mould resting on a non-porous plate. The mould is completely filled and the surface of the mix is then struck off level with the top of the mould. In filling the mould, the blade of a suitable trowel or spatula is used. The mould after filling may be lightly shaken to the extent necessary for expelling the air. Clean appliances are used for gauging and the temperature of the materials and that of the rest room at the time when the above operations are being performed is $27 \pm 2^{\circ}\text{C}$.

A-3.2 Determination of Consistency — The test block confined in the mould and resting on the plate is placed under the rod bearing the plunger; the later is then lowered gently into contact with the surface of the test block and quickly released and allowed to sink into it.

Trial mixes shall be made up with varying percentages of solution until the amount necessary for determining the normal consistency as defined above is found. The amount of solution used shall be recorded and expressed as a percentage by weight of the dry calcined magnesite.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mol	mole

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²